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MEMORANDUM

STABILITY OF CERAMICS IN HYDROGEN BETWEEN 4000° AND 4500° F

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SUMMARY

The various reactions that are possible between hydrogen and certain ceramic materials are discussed as well as the means of measuring the extent of such reactions. Powdered carbides, nitrides, borides, and oxides were tested. These materials were heated inductively in a tungsten cup between 4000° and 4500° F for two 1-hour periods under a static hydrogen atmosphere. Weight, pressure, and diffraction pattern changes were observed, and these served to indicate the extent of reaction.

Most of the ceramics, HfC, ZrC, TiC, TaC, NbC, WC, Mo_2C, HfN, ZrN, NbN, ZrB_2, NbB_2, and WB, showed less reaction than the minimum detectable value. However, the ceramics, TiN, TaN, HfB_2, TiB_2, ZrO_2, and Cr_2O_3, apparently reacted to a measurable extent with hydrogen. Reactions of SiC, VC, and TaB_2 with hydrogen were not determinable because of their incompatibility with the tungsten container.

INTRODUCTION

Recently, the use of ceramic materials at high temperatures has become of great interest. Most applications of such refractory ceramics would involve the presence of some sort of gaseous environment. The extent of reaction between these ceramics and gases at such temperatures cannot be satisfactorily calculated from thermodynamic data because of the large extrapolations that are necessary.

This report presents the results of a preliminary investigation of the reactions between hydrogen gas and powdered refractory carbides, nitrides, borides, and oxides between about 4000° and 4500° F. The tests were conducted in a static system to ascertain roughly which reactions occur. Three things should be considered in a discussion of such reactions: (1) possible types that can occur, (2) reliability of the methods used for their measurements, and (3) the actual extent of such reactions.

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EXPERIMENTAL PROCEDURE

Materials

The ceramic powders used were: hafnium carbide, HfC; zirconium carbide, ZrC; titanium carbide, TiC; tantalum carbide, TaC; niobium carbide, NbC (CbC); vanadium carbide, VC, tungsten carbide, WC; molybdenum carbide, Mo_2C; silicon carbide, SiC; hafnium nitride, HfN; zirconium nitride, ZrN; titanium nitride, TiN; tantalum nitride, TaN; niobium nitride, NbN (CbN); hafnium diboride, HfB_2; zirconium diboride, ZrB_2; titanium diboride, TiB_2; tantalum diboride, TaB_2; niobium diboride, NbB_2 (CbB_2); tungsten boride, WB; zirconium dioxide, ZrO_2; and chromium sesquioxide, Cr_2O_3.

Very few of the ceramics used can be obtained in a high state of purity; the samples used were in general above 90 percent pure, as indicated in table I. The percent purity given for each ceramic material is the combined percents of the major metal and major nonmetal that were found by chemical analysis. The atomic ratio of the nonmetal to metal for each material corresponds to within 5 percent of the stoichiometric value indicated in the first column of each table.

Although the samples used were not 100 percent pure, their X-ray diffraction patterns were in good agreement with those reported for these ceramics in the literature (refs. 1 to 9; see also table I).

Apparatus

The diagram of the reaction chamber is given in figure 1. The ceramic powder was contained in a tungsten cup (3/4 in. high, 3/4-in. O.D., and 1/16-in. wall), which was supported on three pointed tungsten rods (about 3-in. long and 1/32-in. diam.). The tungsten rods were held in a stainless-steel support by means of individual setscrews. The tungsten cup was heated inductively my means of a 7-turn coil ($1\frac{1}{4}$-in. I.D.) of flattened copper tubing (originally 1/4-in. O.D.). The power supply was a 15-kilowatt spark-gap unit ($7\frac{1}{2}$-kw maximum output; operating between 100 and 400 kc).

The tungsten cup and ceramic sample were surrounded by a quartz tube (12 in. long and 1-in. I.D.) that served as the chamber for containing the gaseous hydrogen used for testing. The quartz chamber was surrounded by a Plexiglas jacket through which water was circulated at the rate of

6 gallons per minute. The induction coil situated within the cooling jacket was insulated from the circulating water by a coating of Glyptal. The quartz chamber was connected to a manifold so that it could be evacuated and then filled with hydrogen; a mercury manometer was used to measure the pressure. Under these experimental conditions a sample could be heated to 4800° F. However, under the stresses caused by the temperature gradients associated with such a temperature, the quartz tube would usually break; consequently, most tests were performed at lower temperatures.

The temperature was measured by an optical pyrometer focused through a reflecting prism and an optical flat onto the top edge of the tungsten cup containing the sample. The reading was corrected for absorption of the optical system (prism and flat), the emissivity of the tungsten (0.436 at 0.65 μ, ref. 10), and the difference between the temperature of the cup and that of the sample. (See the appendix for details.) With such corrections, the temperatures reported should be within $\pm 30^{\circ}$ F of the true temperature.

Procedure

One gram of the ceramic powder to be tested was weighed into a tungsten cup of known weight. The tungsten cup was then placed on the three tungsten rods in the apparatus. The system was first evacuated (to about 0.1 mm of Hg) and then flushed with hydrogen. The system (capacity of 1653 ml) was reevacuated to about 0.1 millimeter of mercury; hydrogen was finally admitted to a known pressure (about 300 mm Hg). The hydrogen was purified and dried by means of a Deoxo unit and a liquid nitrogen trap.

The sample was then heated at a reduced temperature (about 500° F below the testing temp.) for 10 minutes, primarily as a safety procedure. This preliminary heating period also served as a means of degassing. The sample was then heated at temperature (4000° to 4500° F) for 1 hour and then allowed to cool. The pressure was recorded during the entire run. The sample was reweighed and then reheated in the same manner as before, but without the initial 10-minute preheating period. The sample was weighed again after the second hour; it was then removed from the tungsten cup so that the cup could be reweighed. In some cases, when reactions were suspected, a gas sample was taken after a run and its infrared spectrum was determined; in none of the runs was a reaction product ever detected by this means.

Finally, an X-ray diffraction pattern was taken of the tested sample and compared with the pattern of the original material. For cases in which the powder had sintered, the pattern was taken of the upper face

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of the pellet to avoid lines due to tungsten or products from a reaction between tungsten and the sample. The data are summarized in table II. The pressure changes given are those obtained by subtracting the pressure before heating from the pressure after the entire system had again reached room temperature.

DISCUSSION

The extent to which reaction occurs between ceramic materials and hydrogen cannot be discussed without first considering the possible kinds of reactions that can occur and the reliability of methods used in their measurement.

Possible Reactions

The following four general types of reactions between hydrogen and ceramics can be postulated:

(I) Partial reduction

$$MX + H_2 \rightarrow M_2X + \text{hydride of } X\uparrow$$
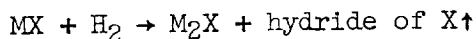
(II) Complete reduction with possible vaporization of resultant metal

$$MX + H_2 \rightarrow M(\uparrow) + \text{hydride of } X\uparrow$$
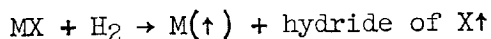
(III) Metal hydride formation

$$MX + H_2 \rightarrow MH_2 + \text{hydride of } X\uparrow$$
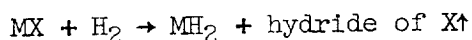
(IV) Addition reaction with possible side products

$$MX + H_2 \rightarrow MXH_2$$
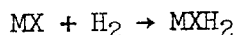
where M represents the metal and X represents either carbon, nitrogen, boron, or oxygen, but not necessarily a single atom.

Reactions (I) and (II) are the most probable ones. Reaction (III) is not likely to occur, since metal hydrides are unstable at the temperatures of this investigation. Reaction (IV) is also improbable, because compounds such as MXH_2 are unknown.

At high temperatures (4000° to 4500° F) the nonmetal hydrides (methane, ammonia, and diborane) could decompose, since they are unstable with respect to their elements. Thus, hydrogen could effectively

be acting only as a catalyst in reactions (I) and (II):

$$2MX \xrightarrow{H_2} M_2X + X$$

$$MX \xrightarrow{H_2} M + X$$
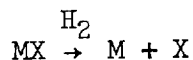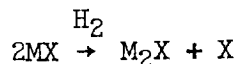
Reliability of Measurement

The reactions, in general, are accompanied by changes in (1) the weight of the sample, (2) the pressure of the system, (3) the composition of the gas, (4) the chemical composition of the sample, and (5) the crystalline structure of the sample.

Measurement of such changes may be in slight error because of degassing or vaporization. The material that vaporizes may be either the ceramic, its decomposition products, or a substance formed by the reaction of the ceramic with the tungsten container. However, the weight loss always set a value for the maximum amount of reaction that could have occurred. If reaction (II) is assumed to occur with no metal volatilization, the maximum percent reacted is equal to

$$\frac{(\text{Weight loss})(\text{Mol. wt. of ceramic})(100)}{(\text{Sample weight})(\text{At. wt. of nonmetal})(\text{Subscript of nonmetal in formula})}$$

Such a calculation has been made for the second weight loss of each sample (table II), because the second heating is more nearly free of degassing effects. These values would be the same order of magnitude if reaction (I) were assumed. The possible experimental error in weight loss (± 3 mg) causes the minimum detectable amount of reaction to be from 1 to 5 percent, depending upon the molecular weight of the ceramic.

Measurement of pressure changes sometimes supplements weight-loss data. Of course, some reactions can occur with no accompanying pressure change; for example,

$$ZrO_2 + 2H_2 \rightarrow Zr + 2H_2O \text{ (gaseous)}$$
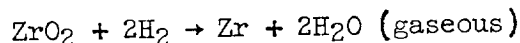
Other reactions can produce positive or negative changes depending upon the materials involved. A completely reacted sample would produce a pressure change (when one can occur) of about 100 millimeters of mercury. Thus, in the present investigation the small pressure changes during the second heating tend to indicate that no severe reaction occurred between any of the ceramic materials and hydrogen. The pressure changes observed (several mm Hg) can easily be attributed to small temperature fluctuations.

The use of the infrared spectrum was chosen as the most convenient method of gas analysis. The infrared, of course, cannot detect the presence of nitrogen gas; but any gas such as CH_4, NH_3, or B_2H_6 is easily detected and identified when present to the extent of several percent. If such gases had been found, the existence of reaction would have been proved.

Detection of small changes in composition of the sample by chemical means was not attempted, since the analyses of many of the original materials were not known with certainty.

A method that is more sensitive to composition changes than chemical analysis is the determination of changes in crystal structure or in lattice parameter. A new pattern, since it is detectable only when about 5 percent or more of a new phase is present, indicates that reaction has taken place to the extent of several percent. This reaction could, of course, be simple decomposition of the ceramic and not involve the hydrogen.

Extent of Reactions and Conclusions

In general, all the ceramics tested showed little or no reaction with a static hydrogen atmosphere in the range 4000° to 4500° F. In general, the carbides and borides showed slight reaction with the tungsten container, while the nitrides were compatible.

Carbides. - The weight losses after the second heating for HfC, ZrC, TiC, TaC, NbC, WC, and Mo_2C were less than the possible experimental error. These carbides are therefore classed as unreactive with static hydrogen. The fact that no negative pressure changes occurred supports this conclusion. Except for WC, there was no large change in diffraction pattern; the appearance of W_2C lines in the pattern for the WC sample is attributable to the reaction of WC with the tungsten cup. The extremely small changes in the lattice parameter of other carbides can easily be due to slight contamination with tungsten. Slight reaction between most of the carbides and tungsten is indicated by the change in the weight of the cup. The tungsten reaction with VC and SiC was so severe that any possible reaction between these materials and hydrogen was masked.

Nitrides. - The small weight losses for samples of HfN, ZrN, and NbN upon the second heating indicate that these materials are nonreactive with hydrogen. The changes in the lattice parameter for these materials are small enough to be ignored. The weight-loss data indicate that TiN and TaN reacted with hydrogen to the extent of 4 to 10 percent. This is supported in the case of TaN by the large change in diffraction pattern: hexagonal to face-centered cubic.

Borides. - Nonreactivity of ZrB_2, NbB_2, and WB with hydrogen is indicated by the small weight changes and supported by negligible pressure changes. Additional lines were found in the diffraction pattern of the NbB_2 sample. These lines, which correspond to an orthorhombic structure, must be attributed to a surface reaction, since the weight loss sets the maximum amount of reaction of the bulk at 0.8 percent. As shown by weight-loss data, the samples of HfB_2 and TiB_2 apparently reacted with hydrogen to the extent of about 8 and 2 percent, respectively. The reactivity of HfB_2 is substantiated by the presence of lines for HfB in the diffraction pattern of the tested specimen. Any reaction that may have occurred between hydrogen and TaB_2 was masked because of the incompatibility of TaB_2 with the tungsten cup.

Oxides. - From weight-loss data it is concluded that ZrO_2 reacted slightly with hydrogen. One extra line in the diffraction pattern supports this conclusion.

The sample of Cr_2O_3 volatilized very rapidly upon heating and condensed on the walls of the quartz reaction chamber. A distinctive yellow powder was detectable in the condensed Cr_2O_3; the five extra diffraction lines were shown to be due to this yellow powder. The amount of this powder and intensity of the lines indicated that reaction had occurred at least to the extent of 5 percent.

Lewis Research Center
    National Aeronautics and Space Administration
        Cleveland, Ohio, December 8, 1958

APPENDIX - TEMPERATURE CALIBRATION

Correction for the absorption of the optical system (prism and flat) was made by calibrating the pyrometer against a standard tungsten lamp with the prism and optical flat in the light path. The calibration curve is given in figure 2. This also corrects for any systematic error in the pyrometer itself. The error in the pyrometer was found to be very small through a calibration without the use of the prism and the flat.

Although corrections for the absorption of the optical system and for the emissivity of the tungsten cup give the proper temperature of the cup, there remains the possibility that the sample inside the cup may be at a slightly lower temperature than the cup. This was checked by determining the melting point of a sheet of molybdenum in the experimental apparatus used for testing the ceramic powders. The molybdenum was found to melt when the edge of the tungsten cup read $4200^{\circ} \pm 5^{\circ}$ F (uncorrected), corresponding to 4858° F corrected for absorption and emissivity. The accepted melting point of molybdenum is $4748^{\circ} \pm 18^{\circ}$ F (ref. 11). Thus, a 110° F temperature difference existed between the tungsten cup and the sample at the cup temperature of 4858° F. It was then assumed that the temperature correction was proportional to the difference between cup temperature and room temperature and that a correction thus calculated would be fairly accurate in the 4000° to 4500° F range:

$$\text{Correction} = m(T - 100)$$

$$110 = m(4858 - 100)$$

$$\therefore m = \frac{110}{4758} = 0.023$$

With these corrections the temperatures listed in table II are believed to be correct to within $\pm 30^{\circ}$ F.

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TABLE I. - SOURCE AND PURITY OF CERAMIC MATERIALS

Material	Source	Purity, percent	Extra lines (all weak)	Crystal structure	Diffraction data				References (parameters are from underlined ref.)
					Lattice parameters				
					Experimental		Literature		
					a	c	a	c	
HfC	The Carborundum Co.	96.3	0	F.c.c.	4.62	-----	4.64	-----	_4_,8
ZrC	Norton Co.	----	0	F.c.c.	4.68	-----	4.685	-----	1,_4_,8
TiC	American Electro Metal Div.	95.8	0	F.c.c.	4.31	-----	4.32	-----	1,_8_
TaC	Charles Hardy, Inc.	----	0	F.c.c.	4.43	-----	4.455	-----	1,_8_
NbC	American Electro Metal Div.	----	0	F.c.c.	4.47	-----	4.461	-----	1,_8_
VC	----	82.6	5	F.c.c.	4.16	-----	4.16	-----	1,_8_,9
WC	Moly. Corp. of America	----	4	Hexagonal	2.90	2.84	2.900	2.831	1,_8_,9
Mo₂C	----	----	0	Hexagonal	2.99	4.72	3.002	4.724	1,_8_
SiC	The Carborundum Co.	----	3	Hexagonal	"d" Values are within 2% of literature		3.073	37.70	1,_9_
HfN	The Carborundum Co.	96.6	0	F.c.c.	4.51	-----	4.52	-----	_4_
ZrN	Metal Hydrides Inc.	----	0	F.c.c.	4.58	-----	4.567	-----	_4_,8
ZrN (lump)	American Electro Metal Div.	97.9	2	F.c.c.	4.57	-----			
TiN		98.1	1	F.c.c.	4.24	-----	4.23	-----	5,_8_(1 in error)
TaN		98.9	1	Hexagonal	5.17	2.92	5.185	2.908	
				Hexagonal	3.04	5.16	2.93	5.45	_6_(8 and 1 in error)
NbN	Cooper Metallurgical Associates	78.0	6	F.c.t.	4.35	4.29	4.38 (4.31) (For NbN₀.₇₅)	4.31	1,_7_
HfB₂	The Carborundum Co.	93.3	3	Hexagonal	3.13	3.47	3.141	3.470	2,_4_,8
ZrB₂	American Electro Metal Div.	98.1	8	Hexagonal	3.17	3.55	3.170	3.533	2,_3_,8
TiB₂		94.3	0	Hexagonal	3.01	3.22	3.028	3.228	2,5,_8_
TaB₂		96.0	3	Hexagonal	3.08	3.23	3.088	3.241	2,_8_
NbB₂		94.0	0	Hexagonal	3.08	3.27	3.086	3.306	2,_8_
WB	Cooper Metallurgical Associates	97.8	0	Tetragonal	2.78	15.84	3.115	16.93	_8_,9
ZrO₂	The Carborundum Co.	C.P.	0	Monoclinic	"d" Values are within 1/2% of literature		5.17 (b = 5.26; β = 80°10')	5.30	1,_9_
ZrO₂ (lump)	Norton Co.	95.0	0	Monoclinic (weak) Cubic	5.11	-----	5.102 (Stabilized by MgO)		_1_,9
Cr₂O₃	Elmer and Amend	99.9	0	Hexagonal (rhombohedral)	"d" Values are within 1/2% of literature		5.361 (α = 55°6')		1,_9_

TABLE II. - HIGH TEMPERATURE REACTION DATA

Material	Temperature, °F	Pressure change, mm Hg	Weight change of specimen and cup, mg	Maximum reaction calculated from weight loss, percent	Weight change of cup, mg	Change in lattice parameter, percent	Diffraction data: Lines that disappeared	Diffraction data: New lines that appeared
HfC	4[illegible] (First run) 4250 (Second run)	-1 0	-1.1 +.7	---	---- 0	---- 0		
ZrC	4[illegible] [illegible]	[illegible] [illegible]	-9.8 -.8	--- *0.7	---- 35.0	---- 0		
TiC	4[illegible] [illegible]	7 5	-15.[illegible] -5.0	--- *1.0	---- [illegible].5	---- -.05		
TaC	[illegible] [illegible]	[illegible] 1	-1[illegible] -2.1	--- *5.4	---- .4	---- -.0[illegible]		
NbC	[illegible] 4[illegible]	5 [illegible]	-7.7 -2.0	--- *1.7	---- [illegible]	---- -.05		
VC	[illegible] [illegible]	40 4	-110.9 -9.4	--- 4.9	---- 60.0	---- -.05	The [illegible] extra lines	
WC	[illegible] [illegible]	5 2	-20.1 -1.3	--- *2.1	---- 51.5	---- ----	WC hexagonal pattern + the 4 extra lines	W_2C hexagonal pattern (ref. 1) a = [illegible] A, c = [illegible] A, + 4 weak lines
Mo_2C	4250 [illegible]	[illegible] 1	-11.[illegible] -2.1	--- *[illegible]	---- [illegible].4	---- -.1		
[illegible]	4[illegible] (15 min) (No reaction line)**	0	-----		----	----		
[illegible]	[illegible] [illegible]	7 5	-7.4 -.7	--- *0.4	---- 3.1	---- 0.5		
[illegible]	[illegible] [illegible]	1[illegible] 1	-4[illegible].7 -[illegible]	--- *[illegible]	---- -5.3	---- 0		
[illegible] (Cup)	[illegible] [illegible]	2 2	-3.0 -.[illegible]	--- *.[illegible]	---- -.[illegible]	---- 1.3	The [illegible] extra lines	
[illegible]	[illegible] [illegible]	2 4	-17.1 -11.7	--- 5.1	---- -1.[illegible]	---- -.05	The [illegible] extra lines	
[illegible]	[illegible] [illegible]	22 3	-81.5 -7.5	--- 1[illegible].4	---- -4[illegible].2	---- ----	[illegible] hexagonal pattern + the [illegible] extra lines	[illegible] a = [illegible] A
NbN	[illegible] [illegible]	2[illegible] 7	-14.4 +1.3	---	---- .[illegible]	---- 0	NbN[illegible] pattern weakened	
HfB_2	[illegible] [illegible]	0 5	-50.1 -8.4	--- 7.6	---- -27.5	---- 0	HfB_2, hexagonal pattern weakened	HfB_2 F.c.c. pattern (ref. 4) a = [illegible] A
ZrB_2	[illegible] [illegible]	[illegible] [illegible]	-104.8 +.3	---	---- .3	---- .[illegible]	[illegible] of the extra lines	1 weak line
TiB_2	[illegible] [illegible]	11 3	-60.4 -[illegible]	--- 1.7	---- 17.4	---- 0		
TaB_2	[illegible] (No reaction line)**	[illegible]	-51.1	---	-77.0	----	TaB_2 hexagonal pattern + the 7 extra lines	F.c.c. pattern a = [illegible] A. + [illegible] weak lines
[illegible]	[illegible] [illegible]	14 [illegible]	-[illegible] -1.[illegible]	--- *.6	---- 13.0	---- -.[illegible]		[illegible]
[illegible]	[illegible] [illegible]	[illegible] [illegible]	-5.7 -1.3	--- *[illegible]	---- [illegible]	---- 0		
[illegible] ([illegible])†	[illegible] [illegible]	-1 1	-[illegible].9 -1.4	--- 1.5	---- -[illegible]	---- 0		
[illegible] (Cup)	[illegible] [illegible]	-1 -1	-8.8 -[illegible].1	--- 7.1	---- -[illegible].0	---- 0		1 weak line
[illegible]	[illegible] (No reaction line)**	[illegible]7	-[illegible]		147.7	‡	(‡)	[illegible] lines + [illegible] lines‡

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*[illegible] limit of accuracy.

**[illegible] cup [illegible].

†[illegible].

‡[illegible] of the [illegible] condensed on quartz tube.

Reflecting prism

Bakelite support

Optical flat

T joints

To manifold

H_2O outlet

7-Turn induction coil

Plexiglas jacket

To R. F. generator

Tungsten cup

H_2O inlet

3 Tung-sten rods

Stainless-steel support

Ball joint

Plexiglas

Pyrex

Quartz

O-rings

CD-6462

Figure 1. - Reaction chamber.
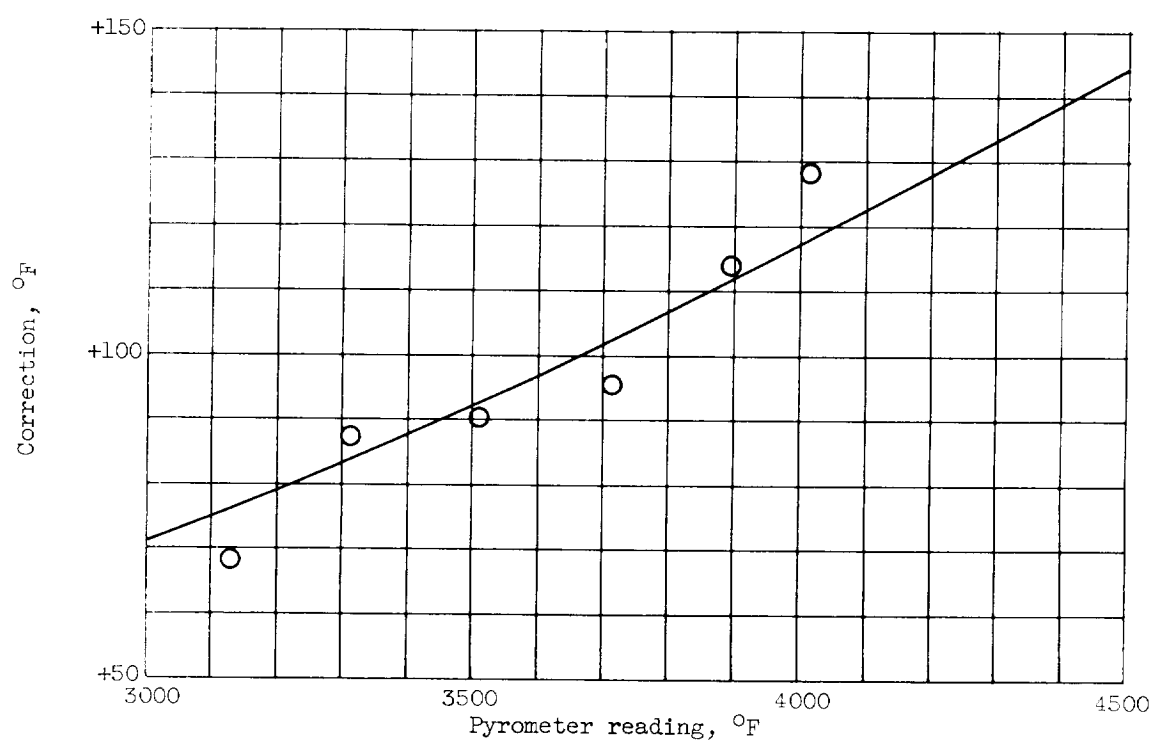
Figure 2. - Calibration curve.

1. Nuclear-Energy
   Systems   (3.1.10)
2. Ceramics   (5.1.5)
3. Materials, Properties -
   Corrosion Resistance
                   (5.2.8)
4. Materials, Properties -
   Thermal   (5.2.11)
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NASA

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(NASA MEMORANDUM 3-5-59E)

The extent of reaction of hydrogen with various carbides, nitrides, borides, and oxides was studied in a static system. Most of these materials, HfC, ZrC, TiC, TaC, NbC, WC, Mo_2C, HfN, ZrN, NbN, ZrB_2, NbB_2, and WB, showed less reaction than the minimum detectable value. However, the data seem to indicate that the ceramics, TiN, TaN, HfB_2, TiB_2, ZrO_2, and Cr_2O_3, had reacted. Reactions of SiC, VC, and TaB_2 were not determinable because of their incompatibility with the tungsten container.

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